

(19)



Europäisches Patentamt
European Patent Office
Office européen des brevets



(11)

EP 0 711 827 A2

(12)

EUROPEAN PATENT APPLICATION

(43) Date of publication
15.05.1996 Bulletin 1996/20

(51) Int Cl⁶ **C11D 17/00, C11D 3/20,
C11D 3/12**

(21) Application number **95307962.1**

(22) Date of filing **07.11.1995**

(84) Designated Contracting States
CH DE ES FR GB IT LI NL SE

(30) Priority **14.11.1994 GB 9422895**

(71) Applicants
• **UNILEVER PLC**
London EC4P 4BQ (GB)
Designated Contracting States
CH DE ES FR GB IT LI NL SE
• **UNILEVER N.V.**
NL-3013 AL Rotterdam (NL)
Designated Contracting States
CH DE ES FR IT LI NL SE

(72) Inventors
• **Edwards, Sara J.**
Unilever Res. Port Sunlight Lab.
Wirral, Merseyside, L63 3JW (GB)

• **Farnworth, Pauline.**
Unilever Res Port Sunlight Lab
Wirral, Merseyside, L63 3JW (GB)
• **Knight, Peter Cory.**
Unilever Res Port Sunlight Lab
Wirral, Merseyside, L63 3JW (GB)
• **Wraige, Douglas.** Unilever Res Port Sunlight Lab
Wirral, Merseyside, L63 3JW (GB)

(74) Representative **FranSELLA, Mary Evelyn**
Unilever PLC
Patent Division
Colworth House
Sharnbrook
Bedford MK44 1LQ (GB)

(54) **Tablet detergent compositions**

(57) A tablet made by compaction of a particulate detergent composition comprising detergent active and aluminosilicate detergency builder is characterised by containing particles which are or contain a major pro-

portion of a highly water-soluble material. This improves disintegration of the whole tablet and dissolution of its soluble ingredients when the tablet is added to water.

EP 0 711 827 A2

Description

The present invention relates to detergent compositions in the form of tablets of compacted detergent powder

Detergent compositions in tablet form are known in the art, as discussed below, and some products are now on the market. Tablets have several advantages over powdered products: they do not require measuring and are thus easier to handle and dispense into the washload, and they are more compact, hence facilitating more economical storage

Detergent tablets are described, for example, in GB 911204 (Unilever), US 3953350 (Kao), JP 60-015500A (Lion), JP 60-135497A (Lion) and JP 60-135498A (Lion), and are sold commercially in Spain

Detergent tablets are generally made by compressing or compacting a detergent powder, which includes detergent active and detergency builder. EP-A-522756 explains that difficulty has been found in providing tablets which have adequate strength when dry, yet disperse and dissolve quickly when added to wash water. The problem has proved especially difficult with compositions containing insoluble aluminosilicate as detergency builder

This prior document teaches that at least some particles of the composition should be coated with a binder which helps to hold the tablet together and allows a tablet to be made using a lower compaction pressure. The binder can also function as a disintegrant. Compositions exemplified in this document include 35% aluminosilicate builder together with 27% detergent active, approximately 20% of non-detergent water soluble materials and 3% of organic binder

Surprisingly we have now found that a considerable improvement in the speed of disintegration in water can be obtained by including a quantity of a highly water-soluble citrate in the tablet composition

Accordingly, the present invention provides a tablet of a compacted particulate detergent composition comprising

from 5% to 50% by weight of one or more detergent-active compounds, and

from 15% to 60% by weight of aluminosilicate detergency builder,

characterised in that the composition includes particles which contain, as a majority of their own weight, a citrate which has a solubility in deionised water at 20°C of at least 50 grams per 100 grams of water

The said particles may consist exclusively of said citrate or they may contain it in a mixture with other material provided that citrate of the specified solubility provides at least 50% by weight - i.e. a majority of the weight - of these particles

The citrate of the specified solubility may well be at least 7%, better at least 12% of the composition. The said particles may provide citrate of the specified solubility in an amount which is at least 7% or 12% of the composition

In some forms of this invention the composition as a whole comprises from at least 15% by weight, better at least 25% or 30% up to 75% by weight of material, other than detergent active, which has a water solubility of at least 2 grams per 100 grams of distilled water at 20°C

In another aspect this invention provides a tablet of a compacted particulate detergent composition comprising

from 5% to 50% by weight of one or more detergent-active compounds, and

from 15% to 60% by weight of aluminosilicate detergency builder,

characterised in that the composition comprises from 25% to 75% by weight of material, other than said detergent active, with a solubility in deionised water at 20°C of at least 2 grams per 100 grams of water

DETERGENT-ACTIVE COMPOUNDS

The total amount of detergent-active material in a tablet of this invention is suitably from 5 to 50 wt%, and is preferably from 5 or 9 wt% up to 40 or 50 wt%. Detergent-active material present may be anionic (soap or non-soap), cationic, zwitterionic, amphoteric, nonionic or any combination of these

Anionic detergent-active compounds may be present in an amount of from 0.5 to 40 wt%, preferably from 2 or 4% to 30 or 40 wt%

Synthetic (i.e. non-soap) anionic surfactants are well known to those skilled in the art. Examples include alkylbenzene sulphonates, particularly sodium linear alkylbenzene sulphonates having an alkyl chain length of C₈-C₁₅, olefin sulphonates, alkane sulphonates, dialkyl sulphosuccinates, and fatty acid ester sulphonates

Primary alkyl sulphate having the formula



in which R is an alkyl or alkenyl chain of 8 to 18 carbon atoms, especially 10 to 14 carbon atoms and M⁺ is a solubilising cation, is commercially significant as an anionic detergent active. It is frequently the desired anionic detergent and may provide 75 to 100% of any anionic non-soap detergent in the composition

In some forms of this invention the amount of non-soap anionic detergent lies in a range from 0.5 to 15 wt% of the

tablet composition

It may also be desirable to include one or more soaps of fatty acids. These are preferably sodium soaps derived from naturally occurring fatty acids, for example, the fatty acids from coconut oil, beef tallow, sunflower or hardened rapeseed oil.

Suitable nonionic detergent compounds which may be used include, in particular, the reaction products of compounds having a hydrophobic group and a reactive hydrogen atom, for example, aliphatic alcohols, acids, amides or alkyl phenols with alkylene oxides, especially ethylene oxide, ether alone or with propylene oxide.

Specific nonionic detergent compounds are alkyl (C_{8-22}) phenol-ethylene oxide condensates, the condensation products of linear or branched aliphatic C_{8-20} primary or secondary alcohols with ethylene oxide, and products made by condensation of ethylene oxide with the reaction products of propylene oxide and ethylene-diamine. Other so-called nonionic detergent compounds include long-chain amine oxides, tertiary phosphine oxides, and dialkyl sulphoxides.

Especially preferred are the primary and secondary alcohol ethoxylates, especially the C_{12-15} primary and secondary alcohols ethoxylated with an average of from 5 to 20 moles of ethylene oxide per mole of alcohol.

In certain forms of this invention the amount of nonionic detergent lies in a range from 4 to 40%, better 4 or 5 to 30% by weight of the composition.

The nonionic detergent-active compounds are preferably concentrated in discrete domains. Since the nonionic detergent compounds are generally liquids, these domains are preferably formed from any of the well known carriers in the detergent business impregnated by nonionic detergent-active compound. Preferred carriers include zeolite, zeolite granulated with other materials, for example Wessalith CS (Trade Mark), Wessalith CD (Trade Mark) or Vegabond GB (Trade Mark), sodium perborate monohydrate, Burkeite (spray-dried sodium carbonate and sodium sulphate as disclosed in EP 221776 (Unilever)).

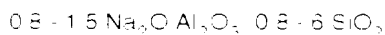
Nonionic detergent-active compounds may optionally be mixed with materials which make the granules slow wetting and/or prevent the nonionic leaching out into the main tablet matrix. Such materials may suitably be fatty acids, especially lauric acid.

The present invention may be applied with compositions which contain more nonionic detergent than non-soap anionic detergent (if any). In compositions of such character, we have found that a weight ratio of nonionic detergent to non-soap anionic detergent in the range 95:5 to 80:20 has been found to give faster dissolution of tablets than does a mixture with a greater proportion of the anionic detergent.

DETERGENCY BUILDERS

The detergent tablets of the invention contain one or more detergency builders. One builder which must be present is an aluminosilicate. Alkali metal aluminosilicates are strongly favoured as environmentally acceptable builders. However, tablets containing alkali metal aluminosilicates as builders appear to have a particular tendency to exhibit disintegration and dispersion problems.

Alkali metal (preferably sodium) aluminosilicates are incorporated in amounts of from 15 to 60% by weight (anhydrous basis) of the composition, and may be either crystalline or amorphous or mixtures thereof, having the general formula



These materials contain some bound water and are required to have a calcium ion exchange capacity of at least 50 mg CaO/g. The preferred sodium aluminosilicates contain 1.5-3.5 SiC_2 units (in the formula above). Both the amorphous and the crystalline materials can be prepared readily by reaction between sodium silicate and sodium aluminate as amply described in the literature.

Suitable crystalline sodium aluminosilicate ion-exchange detergency builders are described, for example, in GB 1429143 (Procter & Gamble). The preferred sodium aluminosilicates of this type are the well known commercially available zeolites A and X, and mixtures thereof. Also of interest is the novel zeolite P described and claimed in EP 334070 (Unilever).

Other builders may also be included in the detergent tablet of the invention as necessary or desired. Water-soluble builders may be organic or inorganic. Inorganic builders that may be present include alkali metal (generally sodium) carbonate, while organic builders include polycarboxylate polymers, such as polyacrylates, acrylic maleic copolymers, and acrylic phosphonates, monomeric polycarboxylates such as citrates, gluconates, oxydisuccinates, glycerol mono-, di- and trisuccinates, carboxymethyloxysuccinates, carboxymethyloxymalonates, dipicolinates, hydroxyethyliminodiacetates, and organic precipitant builders such as alkyl- and alkenylmalonates and succinates, and sulphonated fatty acid salts.

Especially preferred supplementary builders are polycarboxylate polymers, more especially polyacrylates and acrylic maleic copolymers, suitably used in amounts of from 0.5 to 15 wt%, especially from 1 to 10 wt%, and monomeric polycarboxylates, more especially citric acid and its salts.

The total amount of detergency builder including aluminosilicates will generally not exceed 80% by weight of the composition

Preferred tableted compositions of the invention preferably do not contain more than 5 wt% of inorganic phosphate builders and are desirably substantially free of phosphate builders. However, tableted compositions containing some phosphate builder are also within the scope of the invention

HIGHLY WATER SOLUBLE CITRATE

This citrate has a solubility of at least 50 grams per 100 grams of water at 20°C. This is an exceptionally high solubility, many materials which are classified as water soluble are less soluble than this. Strongly preferred is sodium citrate dihydrate which has a water solubility of 72 grams of solid in 100 grams of water at 20°C.

By contrast the solubilities of some other common materials at 20°C are

Material	Water Solubility (g/100g)
Sodium chloride	36
Sodium sulphate decahydrate	21.5
Sodium carbonate anhydrous	8.0
Sodium percarbonate anhydrous	12
Sodium perborate anhydrous	3.7

Preferably this highly water soluble material is incorporated as particles of the material in a substantially pure form (i.e. each such particle contains over 95% by weight of the material). However, particles containing 50 to 95% of such material, better at least 70% of such material, may be utilised.

As the citrate dissolves it leads to a transient local increase in ionic strength which can assist disintegration of the tablet by preventing nonionic detergent from swelling and inhibiting dissolution of other materials. A significant form of this invention is concerned with tablets containing at least 5% by weight of one or more nonionic detergent-active compounds.

The particles containing the highly water-soluble citrate will generally be mixed with other particles containing the detergent active, aluminosilicate and other constituents of the composition, to provide the overall composition which is compacted into tablets.

Other, non-detergent water-soluble materials may well be included in the composition, usually to perform some function other than tablet disintegration although they may assist this.

As indicated above some detergency builders are water-soluble materials. Some bleaches are water-soluble, notably sodium perborate.

The particulate composition preferably includes a binder material. Preferred is that at least some of the particles of the detergent composition are individually coated with the binder material. Then, when the composition is compacted, this coating serves as a binder distributed within the composition. The binder may be distributed throughout the tablet or a discrete region of the tablet.

It is strongly preferred that the binder is water-soluble and that it serves as a disintegrant by disrupting the structure of the tablet when the tablet is immersed in water, as taught in our EP-A-522766.

Use of a binder helps to hold the tablet together, thus enabling it to be made using a lower compaction pressure and making it inherently more likely to disintegrate well in the wash liquor. If the binder is also a material that causes disruption when contacted with water, even better disintegration properties may be achieved.

It is preferred that the binder material should melt at a temperature of at least 35°C, better 40°C or above, which is above ambient temperatures in many temperate countries. For use in hotter countries it will be preferable that the melting temperature is somewhat above 40°C, so as to be above the ambient temperature.

For convenience the melting temperature of the binder material should be below 80°C.

Preferred binder materials are synthetic organic polymers of appropriate melting temperature, especially polyethylene glycol. Polyethylene glycol of average molecular weight 1500 (PEG 1500) melts at 45°C and has proved suitable. Polyethylene glycol of higher molecular weight, notably 4000 or 6000, can also be used.

Other possibilities are polyvinylpyrrolidone, and polyacrylates and water-soluble acrylate copolymers.

The binder may suitably be applied to the particles by spraying, e.g. as a solution or dispersion. The binder is preferably used in an amount within the range from 0.1 to 10% by weight of the tablet composition, more preferably the amount is at least 1% or even at least 3% by weight of the tablets. Preferably the amount is not over 5% or even 6% by weight.

BLEACH SYSTEM

Tableted detergent compositions according to the invention may contain a bleach system. This preferably comprises one or more peroxy bleach compounds, for example, inorganic persalts or organic peroxyacids, which may be employed in conjunction with activators to improve bleaching action at low wash temperatures. If any peroxygen compound is present, the amount is likely to lie in a range from 10 to 25% by weight of the composition.

Preferred inorganic persalts are sodium perborate monohydrate and tetrahydrate, and sodium percarbonate, advantageously employed together with an activator. Bleach activators, also referred to as bleach precursors, have been widely disclosed in the art. Preferred examples include peracetic acid precursors, for example, tetraacetyethylene diamine (TAED), now in widespread commercial use in conjunction with sodium perborate, and perbenzoic acid precursors. The quaternary ammonium and phosphonium bleach activators disclosed in US 4751015 and US 4818426 (Lever Brothers Company) are also of interest. Another type of bleach activator which may be used, but which is not a bleach precursor, is a transition metal catalyst as disclosed in EP-A-458397, EP-A-458398 and EP-A-549272. A bleach system may also include a bleach stabiliser (heavy metal sequestrant) such as ethylenediamine tetramethylene phosphonate and diethylenetriamine pentamethylene phosphonate.

As indicated above, if a bleach is present and is a water-soluble inorganic peroxygen bleach, the amount may well be from 10% to 25% by weight of the composition.

OTHER INGREDIENTS

The detergent tablets of the invention may also contain one of the detergency enzymes well known in the art for their ability to degrade and aid in the removal of various soils and stains. Suitable enzymes include the various proteases, cellulases, lipases, amylases, and mixtures thereof, which are designed to remove a variety of soils and stains from fabrics. Examples of suitable proteases are Maxatase (Trade Mark), as supplied by Gist-Brocades N.V., Delft, Holland, and Alcalase (Trade Mark), and Savinase (Trade Mark), as supplied by Novo Industri A/S, Copenhagen, Denmark. Detergency enzymes are commonly employed in the form of granules or marumes, optionally with a protective coating, in amount of from about 0.1% to about 3.0% by weight of the composition, and these granules or marumes present no problems with respect to compaction to form a tablet.

The detergent tablets of the invention may also contain a fluorescer (optical brightener), for example, Tinopal (Trade Mark) DMS or Tinopal CBS available from Ciba-Geigy AG, Basel, Switzerland. Tinopal DMS is disodium 4,4'-bis-(2-morpholino-4-anilino-5-triazin-6-ylamino) stilbene disulphonate, and Tinopal CBS is disodium 2,2'-bis-(phenyl-5'-yl) disulphonate.

An antifoam material is advantageously included in the detergent tablet of the invention, especially if the tablet is primarily intended for use in front-loading drum-type automatic washing machines. Suitable antifoam materials are usually in granular form, such as those described in EP 266363A (Unilever). Such antifoam granules typically comprise a mixture of silicone oil, petroleum jelly, hydrophobic silica and alkyl phosphate as antifoam active material, sorbed onto a porous absorbed water-soluble carbonate-based inorganic carrier material. Antifoam granules may be present in an amount up to 5% by weight of the composition.

It may also be desirable that a detergent tablet of the invention includes an amount of an alkali metal silicate, particularly sodium ortho-, meta- or preferably alkali metal silicates at levels, for example, of 0.1 to 10 wt%, may be advantageous in providing protection against the corrosion of metal parts in washing machines, besides providing some measure of building and giving processing benefits.

Effervescent disintegrants may be incorporated in the tablet composition. This category of materials includes weak acids or acid salts, for example, citric acid, maleic acid or tartaric acid, in combination with alkali metal carbonate or bicarbonates; these may suitably be used in an amount of from 1 to 25 wt%, preferably from 5 to 15 wt%. Further examples of acid and carbonate sources and other effervescent systems may be found in Pharmaceutical Dosage Forms, Tablets, Volume 1, 1989, pages 287-291 (Marcel Dekker Inc., ISBN 0-8247-8044-2).

Further ingredients which can optionally be employed in the detergent tablet of the invention include antiredeposition agents such as sodium carboxymethyl cellulose, straight-chain polyvinyl pyrrolidone and the cellulose ethers such as methyl cellulose and ethyl hydroxyethyl cellulose, fabric-softening agents, heavy metal sequestrants such as EDTA, perfumes, pigments, colorants or coloured speckles, and inorganic salts such as sodium and magnesium sulphate. Sodium sulphate may, if desired, be present as a filler material in amounts up to 40% by weight of the composition, however as little as 10% or less by weight of the composition of sodium sulphate, or even none at all, may be present.

As well as the functional detergent ingredients listed above, there may be present various ingredients specifically for aid in tableting. Binders and disintegrants have already been discussed. Tablet lubricants include calcium, magnesium and zinc soaps, especially stearates, talc, glyceryl benzoate, Myvatex (Trade Mark), TL ex Eastman Kodak, sodium benzoate, sodium acetate, polyethylene glycols, and colloidal silicas, for example, Aerasil (Trade Mark), ex Crostfield Chemicals Ltd.

PARTICLE SIZE AND DISTRIBUTION

A detergent tablet of this invention, or a discrete region of such a tablet, is a matrix of compacted particles. Preferably the particulate composition which is compacted is substantially free of small particles.

More preferably, the composition consists substantially wholly of particles within the size range of 200 to 2000 μm , more preferably from 250 to 1400 μm . It is desirable that not more than 5 wt% of particles should be larger than the upper limit, and not more than 5 wt% should be smaller than the lower limit.

This distribution is different from that of a conventional spray-dried detergent powder. Although the average particle size of such a powder is typically about 300-500 μm , the particle size distribution will include a "fines" (particles $\leq 200 \mu\text{m}$) content of 10-30 wt%.

Such a powder may nevertheless be a suitable starting material for a tablet according to the present invention, although it is strongly preferred that fines are eliminated by sieving before tableting.

While the starting particulate composition may in principle have any bulk density, the present invention is especially relevant to tablets made by compacting powders of relatively high-bulk-density, because of their greater tendency to exhibit disintegration and dispersion problems. Such tablets have the advantage that, as compared with a tablet derived from a low-bulk-density powder, a given dose of detergent composition can be presented as a smaller tablet.

Thus the starting particulate composition may suitably have a bulk density of at least 400 g/litre, preferably at least 500 g/litre, and advantageously at least 700 g/litre.

Granular detergent compositions of high bulk density prepared by granulation and densification in a high-speed mixer/granulator, as described and claimed in EP 340013A (Unilever), EP 352135A (Unilever), and EP 425277A (Unilever), or by the continuous granulation/densification processes described and claimed in EP 367339A (Unilever) and EP 390251A (Unilever), are inherently suitable for use in the present invention.

Most preferred are granular detergent compositions prepared by granulation and densification in the high-speed mixer/granulator (Fukae mixer), as described in the above-mentioned EP 340013A (Unilever) and EP 425277A (Unilever). With some compositions, this process can produce granular compositions satisfying the criteria of particle size distribution given above, without sieving or other further treatment.

The tablet of the invention may be either homogeneous or heterogeneous. In the present specification, the term "homogeneous" is used to mean a tablet produced by compaction of a single particulate composition, but does not imply that all the particles of that composition will necessarily be of identical composition. Indeed it is likely that the composition will contain particles which include highly soluble material, and other, less-soluble particles. The term "heterogeneous" is used to mean a tablet consisting of a plurality of discrete regions, for example, layers, inserts or coatings, each derived by compaction from a particulate composition.

In a heterogeneous tablet, any one or more of the discrete regions may consist essentially of a matrix as defined above. Where two or more such matrices are present in different regions, they may have the same or different particle size ranges; for example, a first region (for example, outer layer) may consist essentially of particles with a relatively wide particle size range (for example, 250 to 1400 μm) while another (inner core) may consist essentially of particles with a relatively narrow particle range (for example, 500 to 710 μm).

It is within the scope of the invention, for a minor proportion of visually contrasting particles not within the size range of the matrix to be present, one example of this being the inclusion of a small proportion of much larger particles. In this embodiment of the invention, the visually contrasting particles should be larger in at least one dimension than the matrix particles. The effect of contrast may be enhanced if the non-matrix particles are of a contrasting shape, for example, noodles. Visual contrast may if desired be further emphasised by the use of a contrasting colour.

PRODUCT CHARACTER

The detergent tablet of the invention may be, and preferably is, formulated for use as a complete heavy-duty fabric washing composition. The consumer then does not need to use a mix of tablets having different compositions.

Although one tablet may contain sufficient of every component to provide the correct amount required for an average washload, it is convenient if each tablet contains a submultiple quantity of the composition required for average washing conditions, so that the consumer may vary the dosage according to the size and nature of the washload. For example, tablet sizes may be chosen such that two tablets are sufficient for an average washload; one or more further tablets may be added if the washload is particularly large or soiled, and one only tablet may be used if the load is small or only lightly soiled.

Alternatively, larger subdivisible tablets representing a single or multiple dose may be provided with scorings or indentations to indicate unit dose or submultiple unit dose size to the consumer and to provide a weak point to assist the consumer in breaking the tablet is appropriate.

The size of the tablet will suitably range from 10 to 160 g, preferably from 15 to 60 g, depending on the conditions of intended use, and whether it represents a dose for an average wash load, or a submultiple of such a dose.

The tablets may be of any shape. However, for ease of packaging they are preferably blocks of substantially uniform cross-section, such as cylinders or cuboids.

TABLETING

Tableting entails compaction of a particulate composition. A variety of tableting machinery is known, and can be used. Generally it will function by stamping a quantity of the particulate composition which is confined in a die.

Tableting may be carried out at ambient temperature or at a temperature above ambient. In order to carry out the tableting at a temperature which is above ambient, the particulate composition is preferably supplied to the tableting machinery at an elevated temperature. This will of course supply heat to the tableting machinery, but the machinery may be heated in some other way also.

For production scale machinery it may be desirable to construct the mould in which tableting occurs so that it incorporates channels for the circulation of liquid at the desired temperature. Alternatively the mould could be surrounded by an electric heating coil, controlled by a temperature sensor in contact with the mould.

The temperature of the particulate composition delivered to the tableting machinery may be regulated by conveying the composition through a tunnel which is heated to the temperature chosen for tableting.

Preparation of the composition may itself generate heat and this may serve to bring the composition to the desired temperature for tableting.

For any given starting composition, the compaction pressure which is used to form the tablets will affect both the strength of the tablets and the length of time for them to disintegrate when put into water. It is an advantage that raising the temperature of tableting allows adequate strength to be achieved with lesser compaction pressures - which may reduce the cost of the tableting machinery.

TESTING

A measure of the strength of tablets is their diametral fracture stress σ calculated from the equation

$$\sigma = \frac{2P}{\pi Dt}$$

where σ is the diametral fracture stress in Pascals, P is the applied load in Newtons to cause fracture, D is the tablet diameter in metres and t is the tablet thickness in metres.

Tablets of the invention preferably have a diametral fracture stress of at least 5 kPa, and more preferably at least 7 kPa.

The speed of disintegration of a detergent tablet can be assessed by means of the following "cage test".

The tablet is weighed, placed in a cage of perforated metal gauze (9 cm x 4.5 cm x 2 cm) having 16 apertures (each about 2.5 mm square) per cm². The cage is suspended in a beaker of demineralised water at 20°C and rotated at 50 rpm. The time taken for the tablet to disintegrate and fall through the gauze (the disintegration time) is recorded (or after 10 minutes, if the tablet has not wholly disintegrated, the residue is determined by weighing after drying).

It will be appreciated that this is a very stringent test, since water temperature and agitation are both much lower than in a real wash situation in a machine with a washload present. Disintegration times under real wash conditions are expected to be shorter.

EXAMPLE 1

A number of tablets were prepared with each of the formulations set out in the following Table.

Composition No	1	2	3
Granulated Components	% by weight		
coconut primary alkyl sulphate	1.72	1.33	1.21
coconut alcohol 3EO	9.25	7.17	6.49
coconut alcohol 6EO	6.17	4.78	4.33
zeolite A24	37.60	29.17	26.41
sap	4.03	3.12	2.83
SCMC	1.26	0.97	0.88
fluorescer	0.27	0.21	0.19

Continuation of the Table on the next page

(continued)

Composition No.	1	2	3
Granulated Components	% by weight		
water	7.62	5.91	5.35
Postdosed Components			
PEG 1500	4.27	4.27	4.27
sodium perborate tetrahydrate	19.53	19.53	19.53
TAED granule	4.2	4.2	4.2
perfume	0.6	0.6	0.6
antifoam	3.47	3.47	3.47
sodium citrate	0.0	15.0	20.0

The production procedure was as follows. The materials listed as "granulated components" were mixed in a Fukae (Trade Mark) FS-100 high speed mixer-granulator. The soap was prepared in situ by neutralisation of fatty acid. The mixture was granulated and densified to give a powder of bulk density greater than 750 g/litre and a mean particle size of approximately 650 μm .

The powder was sieved to remove fine particles smaller than 180 μm and large particles exceeding 1700 μm . The remaining solids were then mixed with the powder in a rotary mixer, after which the perfume was sprayed on, followed by the PEG. The PEG was sprayed at about 30°C with the powder at 35 to 40°C.

TABLET PREPARATION

Detergent tablets were prepared by compaction of the detergent powder formulations at compaction pressure sufficient to produce a diametral fracture stress of at least 5 kPa. The tablets were produced using an Instron Universal Machine to drive a cylindrical steel punch into a cylindrical die. The tablets were of circular cross-section having a diameter of 4.5 cm and a thickness of approximately 2.5 cm.

All tablets were made using a force of 0.5 kN which provided a pressure of 31.4 N cm⁻². The properties of tablets (determined by procedures described earlier) were as follows:

Composition No.	1	2	3
diametral fracture stress (kPa)	12	11	12.8
residue	50%	18%	0%

It can be seen from the above that incorporation of the particles of sodium citrate led to tablets which disintegrated faster, leaving no residue.

EXAMPLE 2

A number of tablets were prepared with each of the formulations set out in the following Table.

Composition No:	1	2	3	4
<u>Granulated Components</u>	% by weight			
coconut primary alkyl sulphate	4.8	1.4	1.3	1.4
coconut alcohol 3EO	--	--	7.1	6.85
coconut alcohol 5EO	11.0	12.4	--	--
coconut alcohol 7EO	--	--	4.7	5.5
zeolite A24	27.8	29.3	29.9	29.3
soap	1.7	2.9	3.0	2.9
SCMC	0.8	0.8	1.0	0.8
Sodium carbonate	1.0	0.3	--	0.6
water	5.3	5.3	5.2	5.3
<u>Postdosed Components</u>				
PEG 1500	4.3	4.3	4.3	4.3
Coated sodium percarbonate	19.5	19.5	19.5	19.5
TAED granule	4.2	4.2	4.2	4.2
perfume	0.6	0.6	0.6	0.6
antifoam	3.6	3.6	3.6	3.6
sodium citrate	15.0	15.0	15.0	15.0

The materials listed as "granulated components" were mixed in a Fukae (Trade Mark) FS-100 high speed mixer-granulator. The soap was prepared in situ by neutralisation of fatty acid.

In the case of compositions 1, 2 and 4 the primary alkyl sulphate, sodium carbonate, much of the water content and a small amount of zeolite were added as preformed granules. In the case of composition 3 the primary alkyl sulphate, nonionic detergent, fatty acid and sodium hydroxide to neutralise the fatty acid were all mixed together before addition to the mixer-granulator.

For all four compositions the mixture was granulated and densified to give a powder of bulk density greater than 750 g/litre and a mean particle size of approximately 650µm.

The powder was sieved to remove fine particles smaller than 180µm and large particles exceeding 1700µm. The remaining solids were then mixed with the powder in a rotary mixer, after which the perfume was sprayed on, followed by the PEG. The PEG was sprayed at about 70°C with the powder at about 35°C.

Detergent tablets were prepared at 40°C, generally as in Example 1, by compaction of the detergent powder formulations at various compaction pressures. The diametral fracture stresses were determined as described earlier.

The disintegration of the tablets in water was tested by the cage test given earlier. The results of these tests are set out below in three tables which show comparison between tablets of composition 1, with a 30/70 ratio of primary alkyl sulphate to nonionic detergent, and tablets of compositions 2, 3 and 4 with a 10/90 ratio.

As can be seen from these tables, tablets of compositions 2, 3 and 4 generally have less residue than tablets of composition 1, with the same or smaller value of diametral fracture stress, thus indicating that the tablets of compositions

2 3 and 4 could be stronger yet dissolve faster than tablets of composition 1

composition	1	2	1	2	1	2	1	2
compaction force (kN)	0.4	0.7	0.5	0.9	1	1.5	2.5	3
density (kg/m ³)	1200	1220	1240	1265	1295	1315	1365	1380
DFS (kPa)	5	6	7.5	9	12	13	16	21
Residue (%)	0	0	50	27	78	50	95	68

composition	1	3	1	3	1	3	1	3
compaction force (kN)	0.4	0.9	0.5	1.5	1	2.1	2.5	3.3
density (kg/m ³)	1200	1240	1240	1290	1295	1340	1385	1390
DFS (kPa)	5	7.5	7.5	10	12	15	16	19
Residue (%)	0	0	50	5	78	45	95	70

composition	4	1	4	1	4	1	4	1
compaction force (kN)	1.1	0.4	1.5	0.5	2	1	2.8	2.5
density (kg/m ³)	1245	1200	1285	1240	1340	1295	1395	1385
DFS (kPa)	4.5	5	7	7.5	10	12	15	16
Residue (%)	0	0	20	50	45	78	85	95

Claims

1. A tablet of a compacted particulate detergent composition comprising

from 5% to 50% by weight of one or more detergent-active compounds and
from 15% to 60% by weight of aluminosilicate detergency builder
characterised in that the composition includes particles which contain from 50 to 100% of their weight of a
citrate with a solubility in deionised water at 20°C of at least 50 grams per 100 grams of water

2. A tablet according to claim 1 wherein the citrate with a said solubility of at least 50 grams per 100 grams of water
provides at least 12% by weight of the tablet composition

3. A tablet according to any one of the preceding claims which contains in addition to said detergent active and
aluminosilicate builder at least 25% by weight of non-detergent material with a solubility in deionised water at
20°C of at least 2 grams per 100 grams of water

4. A tablet according to any one of the preceding claims containing at least 20% by weight of aluminosilicate deter-
gency builder

5. A tablet according to any one of the preceding claims wherein the detergent active comprises

nonionic detergent and non-soap anionic detergent the quantity of nonionic detergent being greater than the
quantity of non-soap anionic detergent

6. A tablet according to claim 5 wherein the ratio by weight of nonionic detergent to non-soap anionic detergent lies
in a range from 20 to 95/5

7. A tablet according to any one of the preceding claims which also includes from 1 to 8% by weight of a polymeric
binder

8. A tablet according to claim 7 wherein the binder is a water-soluble organic polymer and is present in an amount of at least 3% by weight
9. A tablet according to claim 7 or claim 8 wherein the binder melts at a temperature in the range from 35°C to 60°C
10. A process for the preparation of detergent tablets according to any one of claims 7 to 9 comprising the steps of
- (i) preparing a particulate detergent composition including the detergent active and the aluminosilicate
 - (ii) applying the binder as a coating on at least some particles in the detergent composition
 - (iii) compacting the composition into tablets



(19)



Europäisches Patentamt
European Patent Office
Office européen des brevets



(11)

EP 0 711 827 A3

(12)

EUROPEAN PATENT APPLICATION

(88) Date of publication A3
13.11.1996 Bulletin 1996/46

(51) Int Cl⁶ **C11D 17/00, C11D 3/20,
C11D 3/12**

(43) Date of publication A2
15.05.1996 Bulletin 1996/20

(21) Application number **95307962.1**

(22) Date of filing **07.11.1995**

(84) Designated Contracting States
CH DE ES FR GB IT LI NL SE

(30) Priority **14.11.1994 GB 9422895**

(71) Applicants

- **UNILEVER PLC**
London EC4P 4BQ (GB)
Designated Contracting States
GB
- **UNILEVER N.V.**
3013 AL Rotterdam (NL)
Designated Contracting States
CH DE ES FR IT LI NL SE

(72) Inventors

- **Edwards, Sara J.,**
Unilever Res. Port Sunlight Lab.
Wirral, Merseyside, L63 3JW (GB)

- **Farnworth, Pauline,**
Unilever Res Port Sunlight Lab
Wirral, Merseyside, L63 3JW (GB)
- **Knight, Peter Cory,**
Unilever Res Port Sunlight Lab
Wirral, Merseyside, L63 3JW (GB)
- **Wraige, Douglas,** Unilever Res Port Sunlight Lab
Wirral, Merseyside, L63 3JW (GB)

(74) Representative **Fransella, Mary Evelyn**
Unilever PLC
Patent Division
Colworth House
Sharnbrook
Bedford MK44 1LQ (GB)

(54) **Tablet detergent compositions**

(57) A tablet made by compaction of a particulate detergent composition comprising detergent active and aluminosilicate detergency builder is characterised by

containing particles which are or contain a major proportion of a highly water-soluble material. This improves disintegration of the whole tablet and dissolution of its soluble ingredients when the tablet is added to water.

EP 0 711 827 A3



European Patent
Office

EUROPEAN SEARCH REPORT

Application Number
EP 95 30 7962

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
X	US-A-4 642 197 (KRUSE HANS ET AL) * column 6, line 22 - line 37 * * column 6, line 44 - line 49 * * claims *	1	C11D17/00 C11D3/20 C11D3/12
X	EP-A-0 466 484 (UNILEVER PLC ET AL.) * page 5, line 17 - line 39 * * page 5, line 50 * * page 7, line 28 - line 31 * * claims; example 16 *	1,4,7-10	
P,X	WO-A-95 04804 (MARINA PATENT HOLDING SA ET AL.) * example 1 * * claims 1-3,5 *	1,4	
X	EP-A-0 598 586 (UNILEVER PLC ET AL.) * examples * * claims *	1,4	
X	EP-A-0 466 485 (UNILEVER PLC ET AL.) * example D *	1	TECHNICAL FIELDS SEARCHED (Int.Cl.6) C11D
A,D	EP-A-0 522 766 (UNILEVER PLC ET AL.) * page 4, line 27 - line 37 * * page 7, line 3 - line 6 * * claims *	1-10	
The present search report has been drawn up for all claims			
Place of search		Date of completion of the search	Examiner
BERLIN		12 September 1996	Pelli Wablat, B
CATEGORY OF CITED DOCUMENTS		I : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application I : document cited for other reasons & : member of the same patent family, corresponding document	
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document			

EPO FORM 1503 (01/92) (P04C01)